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COLLOIDAL CATALYSTS IN WATER

FINAL REPORT

WARREN T. FORD

September 10, 1992

U. S. ARMY RESEARCH OFFICE

CONTRACT NUMBER DAAL03-89-K-0122

Oklahoma State University
Department of Chemistry
Stillwater, OK 74078

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STATEMENT OF THE PROBLEM STUDIED

Our aims are to understand how the surface structures of colloids affect their activity as catalysts and to design catalysts in aqueous colloids that have general activity for decontamination. We have bound catalysts into the gel phase and onto the surface of copolymer latexes for autoxidation and hydrolysis reactions. These colloidal ion exchange resins could be precipitated to obtain an easily packaged and transported catalyst, and redispersed quickly into water for use in the field. The colloids might also be incorporated into latex coatings having catalytic activity for decontamination.

SUMMARY OF RESULTS

IBA-Catalyzed Hydrolysis of *p*-Nitrophenyl Diphenyl Phosphate

We have found that 0.01-0.8 mg/mL of cationic latex particles and $0.40-5.0 \times 10^{-5}$ M IBA (*o*-iodosobenzoic acid) catalyze hydrolysis of *p*-nitrophenyl diphenyl phosphate at 25 °C and pH 8.0 with a pseudo first-order rate constants of up to 0.064 s^{-1} , which corresponds with a shortest half-life of 12 seconds. The highest second-order rate constant under these conditions exceeds by a factor of about 2 the maximum value reported by Moss for IBA in CTACl micelles, the previous fastest medium for IBA-catalyzed hydrolysis of phosphate esters. So few particles are required that the kinetics can be followed by appearance of UV absorption of *p*-nitrophenoxide ion without excessive loss of signal due to light scattering. The weight concentration of latex particles needed for optimal catalysis is less than the weight concentration of CTACl at its cmc. The most active latexes are 150 nm diameter particles swollen 2.4-10 times their dry volume in water. Less swollen latexes form much less active catalysts.

Extension of the kinetic study to different buffers and to varied pH and ionic strength of the dispersions indicates that latex catalysts closely resemble micellar catalysts. (1) IBA is a turnover catalyst: Good pseudo first-order kinetics are obtained with PNPDP in five-fold excess over IBA. (2) Ultrafiltration of a dispersion containing substrate and buffer but no IBA left no PNPDP detectable by UV absorption in the aqueous phase, indicating that PNPDP is completely bound to the particles. (3) The rate of reaction increases as the number of particles in the dispersion increases at constant IBA and PNPDP concentrations, and then decreases at particle amounts greater than 0.4 mg/mL. In micellar catalysis rates decrease with increasing surfactant concentrations above the cmc due to dilution of reactants in the micelle phase under conditions where most of the IBA and all of the PNPDP are bound. (4) The rate of reaction is highest in the swollen particles having the highest concentration (in mmol/mL) of ion exchange sites, as expected for a second order reaction of IBA with PNPDP. (5) The rate of reaction decreases with increase in concentration of added NaCl and with increase in concentration of the zwitterionic buffer TAPS [N-tris(hydroxymethyl)methyl-3-aminopropanesulfonic acid]. Chloride ion and TAPS anion compete with IBA anion for binding sites in the particles. At pH 8.0 the IBA is effectively all in anionic form in the latexes despite its aqueous phase pK_a of about 7.3. (6) The catalytic activity of the polyelectrolyte having the same repeat unit composition as the latex is less active, but also less sensitive to rate depression by added NaCl because of less effective displacement of IBA anion from polyelectrolyte binding sites than from latex binding sites by chloride ion. Thus qualitatively at present the role of the latex appears to be the same as the role of the micelle in most examples of micellar catalysis, to concentrate the reactants into a small fraction of the volume of the mixture. An important feature of the catalysis results in NaCl solutions is that the particles do not immediately coagulate as many charged colloids do, and substantial catalytic activity remains even in salt water.

Samples of latex have been sent to Dr. William Beaudry of CRDEC to test for IBA-catalyzed hydrolysis of GB. In preliminary experiments following disappearance of GB by

³¹P NMR spectroscopy, he found that at the much higher concentrations of buffer (0.1 M) and substrate (0.01 M) required for NMR spectroscopy that (1) the hydroxyethyl groups of our TAPS buffer displace p-nitrophenoxide from PNPDP to form a new phosphate ester, and (2) borate buffer led to cessation of reaction after partial conversion, apparently due to some reaction between the borate and the IBA. These effects were not important at 10⁻⁵ M substrate and 5 mM buffer in our experiments followed by UV-vis spectroscopy, but we have confirmed Dr. Beaudry's observations for the hydrolysis of higher concentrations of PNPDP in our laboratory. We have now found two new zwitterionic buffers that are well behaved at high concentrations in our lab at pH 8 and pH 9 and have sent them to Dr. Beaudry for further study of latex catalysis of GB hydrolysis.

Characterization of Ion Exchange Latex Particles

The IBA-catalyzed hydrolyses were performed with a series of 7 quaternary ammonium ion latexes having particle sizes of 150±10 nm and charge densities ranging from 1% to 60% of polymer repeat units. *This is the first time cationic latexes have been prepared with the charge density independent of particle size.* Particle size control was achieved using (styrylmethyl)trimethylammonium chloride as a monomer in shot-growth emulsion copolymerizations with styrene, divinylbenzene, and chloromethylstyrene. The chloromethylstyrene units were quaternized with trimethylamine to form the higher charge density latexes.

The latex particles are intriguing in their own right. With light scattering help from our physicist colleague Bruce Ackerson and his students, we have found that the ion exchange latex containing 60 mol % styryltrimethylammonium chloride repeat units and cross-linked with 1% DVB is highly swollen with water as expected, but that static and dynamic light scattering give quite different pictures of the particles. The radius of gyration (R_g , defined as the root mean square distance of a mass element from the center of mass of the particle) of the most highly swollen 75N⁺ particles measured by static light scattering is only 0.58 times the hydrodynamic radius measured by dynamic light scattering (R_h , calculated from the self-diffusion coefficient D and the viscosity η by the Stokes-Einstein equation, $D = kT/6\pi R_h \eta$). For hard spheres the theoretical value of R_g/R_h is 0.775. The experimental results can be explained by a model in which the density of polymer chain segments in the swollen state has a Gaussian distribution about the center of mass. In the model the particles are less swollen in the center and more swollen near the surface, which resembles a concentrated polyelectrolyte. Since a polystyrene cross-linked with 1 mol percent DVB has an average chain length between cross-links of 50 repeat units, there must be many chains that are not cross-linked but are anchored to the polymer network by one chain end. The light scattering study is important, because to understand catalysis, we must understand the particle structures. SLS and DLS measurements in up to 1 M NaCl solutions show greater contraction of the hydrodynamic radius than of the radius of gyration, as expected if the hydrodynamic radius has polyelectrolyte behavior.

Decarboxylation of 6-Nitrobenzisoxazole-3-carboxylate in Latexes

The decarboxylation of the title compound is extremely sensitive to medium effects. The room temperature decarboxylation rate is 10⁸ times faster in HMPA than in water. The reaction has been studied before in a variety of colloidal and polymeric environments with rate enhancements relative to water ranging from 130 by cetyltrimethylammonium bromide micelles to 14,000 by poly(vinylbenzo-18-crown-6 ether). We have studied the decarboxylation kinetics in a variety of latexes and find catalytic rate constant enhancements ranging from 310 in a highly hydrophilic ion exchange latex to 21,000 in a more lipophilic polystyrene latex containing benzyltri-n-butylammonium ion binding sites. *This is the highest such rate enhancement ever observed in an aqueous medium.* A micellar kinetic model and an ion exchange kinetic model give the same values for the catalytic rate constants. The ion exchange model assumes specific ion exchange binding of the substrate, whereas the

micellar model assumes non-specific binding. We are evaluating the validity of the ion exchange model. If correct, it also yields accurate measurements of the ion exchange selectivities of the latexes for all of the anions in the system.

Oxidation of Mercaptans to Disulfides

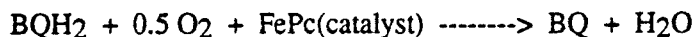
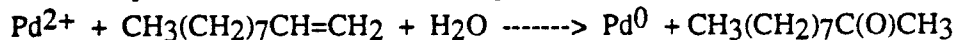
Colloidal silica modified with (3-trimethylammonio)propyl groups, cationic latexes with high and low charge density, and cationic polyelectrolytes have been compared as supports for the cobalt phthalocyaninetetrasulfonate (CoPcTs) catalyzed autoxidation of mercaptoethanol to the disulfide. The cationic polyelectrolytes 2,4-ionene and 3,6-ionene give catalysts >50 times more active than any of the other supported catalysts. Binding of the cationic polyelectrolytes to cationic latexes followed by binding of CoPcTs gives much less active catalysts, whereas a cationic polyelectrolyte and CoPcTs bound to colloidal silica retains most of the activity. The ion exchange latexes are more active than conventional macroporous ion exchange resins.

Oxidation of Lignin Model Compounds

A major industrial problem is the removal of lignin from pulp, a process required for production of high quality paper. For delignification with minimal degradation of cellulose fiber the kraft process, treatment with NaHS at >150 °C, is the best available. The kraft process is strongly alkaline, produces noxious sulfur compounds, and darkens the pulp so that extensive chlorine treatment is needed to produce white paper. Chlorination forms large amounts of toxic organochlorine byproducts. To test the possible use of colloidal catalysts for lignin oxidation we investigated oxidation of the model compounds 3,4-dimethoxybenzyl alcohol, 3,4-dimethoxytoluene, and 2-methoxy-4-methylphenol catalyzed by six different anionic metalloporphyrins and methallophthalocyanines in aqueous dispersions using the mildest possible oxidizing agents. Phthalocyanines are more oxidatively stable than porphyrins. Temperatures of 70-85 °C were necessary to initiate reactions. With dioxygen as the oxidant only CoPcTS was active, and it required pH >9. With hydrogen peroxide only FePcTS was active, and it required pH <4. There was little difference in results in the presence and in the absence of latexes, even though the catalysts bound completely to the particles. These results in colloidal dispersions show promise for oxidations in complex mixtures of solids and water, such as pulp from mechanical breakdown of wood chips and messy field environments that might be encountered in efforts to decontaminate CW agents.

Wacker Oxidation of Alkenes by Dioxygen Catalyzed by Pd(0), Benzoquinone, and Colloidal Iron Phthalocyanine

Colloidal iron phthalocyanine (FePc) is an efficient catalyst for the reoxidation of hydroquinone (BQH₂) to benzoquinone (BQ) in a Wacker oxidation process for the conversion of 1-alkenes to methyl ketones in aqueous N,N-dimethylformamide. The oxidation is complete in 35 minutes at room temperature. A typical catalytic cycle is:



The major variable controlling the activity of this catalytic system is the morphology of the FePc. Samples that appear less crystalline by scanning electron microscopy and by X-ray powder diffraction and have surface areas of about 30 m²/g by BET analysis are highly active; those that are more crystalline and <3 m²/g show little or no activity. The activity is due to the μ -oxo(1) dimer form of FePc, identified by IR spectroscopy.

Oxidations of Alkenes by Aqueous Hydrogen Peroxide Catalyzed by Peroxymolybdate on Latexes

Epoxidation of cyclooctene in aqueous hydrogen peroxide is catalyzed by ammonium molybdate and by phosphomolybdic acid in the absence of organic solvent. Peroxomolybdate bound to 60 nm colloidal anion exchange polymers gave dispersions that were more active than solutions. Typically 1.3 mol % of Mo(VI) bound to colloidal particles catalyzed 90% conversion of 1.50 mmol of cyclooctene with >99% selectivity for cyclooctene oxide by excess hydrogen peroxide in 24 h at 40 °C, but only 12% epoxide was formed in the absence of colloidal particles. Under the same conditions styrene and cyclohexene gave complicated product mixtures, and 1-octene did not react.

Oxidation of Alkenes by Hypochlorite and by Peroxymonosulfate Catalyzed by a Mn(III) Porphyrin on Latexes

A colloidal catalytic method has been devised for the oxidation of styrene to styrene oxide by sodium hypochlorite in water catalyzed by meso-tetra(2,6-dichloro-3-sulfonatophenyl)porphyrinatomanganese(III). The anionic porphyrin is bound to quaternary ammonium ion exchange latexes 60 nm in diameter. The oxidation proceeds to 80% conversion in 30 minutes at room temperature at either pH 10.8 or pH 13.5. The latex-bound porphyrin is at least three times more active than the same porphyrin in aqueous solution. The porphyrin is more stable and the activity lasts longer at pH 13.5. Spectroscopic evidence indicates the presence of an oxo form of the Mn porphyrin in the reaction mixture. The catalyst is selective for styrene and substituted styrenes. Aliphatic alkenes such as cyclooctene and 1-decene react only slowly. By use of more lipophilic quaternary ammonium latexes containing <25% of ionic repeat units and sometimes also containing a lipophilic comonomer such as dodecyl methacrylate, aliphatic alkenes can be oxidized readily at 40 °C to product mixtures containing allylic alcohol, diol, and chlorohydrin in addition to epoxide. With the same latex-bound porphyrin catalysts, potassium monoperoxysulfate (Oxone) is more reactive than NaOCl.

Oxidations of Alkenes by Monoperoxysulfate in Water with No Organic Solvent

The activity peroxymonosulfate in the absence of the porphyrin, in the absence of latex, and in the absence of phase transfer catalysts is only slightly less than that of the more complex colloidal systems. At initial pH 6.8 and 25 °C Oxone selectively epoxidizes cyclooctene, whereas at lower pH it produces mostly cyclooctanediol. Similar results were found with other alkenes. The most important results from this research are that oxone can be used for selective oxidations of alkenes in mixtures of water and organic reactant with no organic solvent, surfactant, phase transfer catalyst, or latex. Simplicity makes these processes appealing for commercial uses.

PATENT MATTERS

Oklahoma State University and the U.S. Government have released rights of U.S. Patents 4,857,493 (Aug. 15, 1989) and 5,011,956 (Apr. 30, 1991) to the inventors, and a royalty-free, non-exclusive license has been issued to the government.

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A. Journals

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B. Conference Proceedings

1. Badley, R. D., Ford, W. T., McEnroe, F. J., and Assink, R. A.; Characterization of Colloidal Silica. Presented at the Chemically Modified Oxide Surfaces Symposium, Midland, MI, June 1989.
2. Srinivasan, S., and Ford, W. T., The Role of Iron(II) Phthalocyanine As a Catalyst in Wacker Oxidation of 1-Decene. American Chemical Society National Meeting, Washington, DC, September 1990, Division of Organic Chemistry.
3. Turk, H.; and Ford, W. T., Epoxidation of Styrene with Aqueous Hypochlorite Catalyzed by a Water-Soluble Mn(III) Porphyrin Bound to Colloidal Anion Exchange Particles. American Chemical Society National Meeting, Washington, DC, Division of Polymer Chemistry, Symposium on Chemical Reactions on Polymers. *Polymer Preprints*, 1990, 31(2), 440.

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7. HariBabu, S.; and Ford, W. T.; Colloidal Catalysts for Autoxidation of Thiols. Presented at the American Chemical Society Division of Colloid and Surface Chemistry National Symposium, Norman, OK, June 1991, at the Gordon Research Conference on Reactive Polymers, Newport, RI, August 1991, and at the US Army Chemical Research Development and Engineering Center: Proceedings of the 1991 Scientific Conference.
8. Zhu, W.; and Ford, W. T.; Oxidation of Alkenes Catalyzed by a Manganese(III) Porphyrin on Cationic Polymer Colloids. Presented at the American Chemical Society Division of Colloid and Surface Chemistry National Symposium, Norman, OK, June 1991; at the Gordon Research Conference on Reactive Polymers, Newport, RI, August 1991; at the American Chemical Society National Meeting, San Francisco, April 1992; Division of Organic Chemistry, and at the US Army Chemical Research Development and Engineering Center: Proceedings of the 1991 Scientific Conference.
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C. Patents

1. Ford, W. T.; Chandran, R. S.; Hassanein, M.; Turk, H., Polymer Latexes Containing Metal Ions and Complexes, U.S. Patent 4,857,493, Aug. 15, 1989.
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D. Ph.D. Dissertations

1. Hayrettin Turk, Oxidation of Phenols and Styrenes in Water Catalyzed by Metal Complexes Bound to Colloidal Particles, Dec. 1989.
2. Sanjay Srinivasan, Oxidation of Alkenes Catalyzed by Colloidal Transition Metal Complexes, Dec. 1990.
3. Weiming Zhu, Oxidations of Alkenes and Lignin Model Compounds in Aqueous Dispersions, May 1992.

SCIENTIFIC PERSONNEL SUPPORTED AND DEGREES AWARDED

Warren T. Ford, 0.5 time July 1 - Aug. 31, 1990, and full time July 1 - July 31, 1991; PI.

S. HariBabu, full time Nov. 6, 1989 - Dec. 31, 1990, Postdoctoral Associate.

Jeng-Jong Lee, full time, Sept. 15, 1991 - Aug. 31, 1992, Postdoctoral Associate.

H. Turk, 0.5 time July 1 - Nov. 30, 1989, Graduate Research Assistant (GRA); Ph.D. December 1989.

Sanjay Srinivasan, 0.5 time June 15, 1989 - June 30, 1990 and Aug. 15 - Oct. 7, 1990, GRA; Ph.D. December 1990.

Weiming Zhu, 0.17 time Oct. 1 - Dec. 31, 1989, 0.5 time Jan. 1, 1990 - Aug. 15, 1990, 0.25 time Aug. 16, 1990 - May 31, 1991, 0.5 time June 1 - Dec. 31, 1991, GRA, Ph.D. May 1992.

Mingyang Zhao, 0.17 time Oct. 1 - Dec. 31, 1989, 0.5 time Jan. 1 - Aug. 15, 1990, 0.25 time Aug. 16, 1990 - May 31, 1991, 0.5 time June 1 - June 30, 1991 - June 30, 1992, GRA.

Hui Yu, 0.5 time June 15 - Aug. 15, 1989, 0.25 time Aug. 16 - Dec. 31, 1989, 0.5 time Jan. 1 - Aug. 15, 1990, 0.5 time May 15, 1991 - June 30, 1992, GRA.

Marietta Bautista, 0.5 time Aug. 16, 1990 - June 15, 1992, GRA.

Savitha Devanathan, 0.5 time May 16 - Aug. 11, 1990, GRA.

Clinton Bryan, 0.5 time July 1 - Aug. 15, 1990 and May 20 - Aug. 3, 1991, GRA.

Marcia Lawson, 0.5 time June 1 - June 30, 1991; GRA.

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With the aims of understanding how the surface structures of colloids affect their activity as catalysts and of designing catalysts in aqueous colloids that have general activity for decontamination, we have bound catalysts for autoxidation and hydrolysis reactions into the gel phase and onto the surface of cationic copolymer latexes. The most effective latexes are water-swollen colloidal anion exchange resins. Iodosobenzoate ion (IBA) bound to latexes is equaled only by IBA in cationic micelles for catalysis of the hydrolysis of phosphate esters. The decarboxylation of 6-nitrobenzisoxazole-3-carboxylate is faster in a latex than in any previous colloidal or polymeric medium. Transition metal complexes, such as a Mn porphyrin, cobalt phthalocyaninetetrasulfonate, and peroxomolybdate, bound to latexes are active for the oxidations of alkenes, thiols, and lignin model compounds with dioxygen, sodium hypochlorite, potassium monoperoxysulfate, and hydrogen peroxide as oxidants. These colloidal ion exchange resins could be precipitated to obtain an easily packaged and transported catalyst, and redispersed quickly into water for use in the field. The colloids might also be incorporated into latex coatings having catalytic activity for decontamination.